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Effects of Thermal Exposure on the Optical Properties of LORD Aeroglaze A276

*David L. Ellis and Donald A. Jaworske
Glenn Research Center, Cleveland, Ohio*

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*David L. Ellis and Donald A. Jaworske
Glenn Research Center, Cleveland, Ohio*

National Aeronautics and
Space Administration

Glenn Research Center
Cleveland, Ohio 44135

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David L. Ellis and Donald A. Jaworske
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

Summary

A lunar outpost will require electrical energy. One potential source is fission surface power where heat from a reactor is converted into electricity utilizing an energy conversion system, and waste heat will need to be rejected from the system. The Second Generation Radiator Demonstration Unit (RDU) is a technology demonstration unit leading towards operational radiators. To approximate the infrared emittance of the lunar outpost radiators, a low-cost coating compatible with the test conditions was sought. LORD Aeroglaze A276 has a similar emittance, but its performance in air and vacuum at the desired operating temperatures was unknown. This study determined that the emittance remained above 0.86 for all conditions tested and that LORD Aeroglaze A276 is a suitable surrogate coating for the Second Generation RDU.

Introduction

A lunar outpost will require ample energy for operations. One source of energy being considered is fission surface power (FSP) where heat from a reactor will be utilized in an energy conversion system to make electricity (Ref. 1). Waste heat from the energy conversion system will need to be rejected to the environment through radiant heat transfer. Hence, any FSP system will require radiators to dissipate waste heat. The Second Generation Radiator Demonstration Unit (RDU) is a technology demonstration program that will ultimately lead to the design and manufacture of radiator panels for use in lunar outpost FSP systems. The Second Generation RDU technology program consists of designing and building a lightweight radiator panel using technologies envisioned for an FSP installation, and testing the panel in a large thermal vacuum chamber. Such technologies include high-efficiency heat exchangers that transfer heat from a closed-loop heat source to lightweight titanium water heat pipes embedded in a high thermal conductivity graphite fiber polymer matrix composite (Ref. 2). Under lunar conditions, the composite face sheets will require a white coating with the optical properties of low solar absorptance and high infrared emittance.

Low solar absorptance is not a requirement for the Second Generation RDU because the panel will not be illuminated while undergoing thermal vacuum testing. High infrared emittance is a requirement needed to reach performance goals. Ideally, the coating selected for use in the Second Generation RDU would be the coating most likely to be used on a lunar outpost radiator. Several studies have been conducted over the years to evaluate the prospect of utilizing commercially available thermal control coatings for the lunar outpost FSP application, including durability under electron exposure, durability under neutron exposure, durability under ultraviolet radiation exposure, wide temperature thermal cycling, and adhesion (Refs. 3 to 7). End-of-life optical properties have been gleaned from the literature for systems that require a 10-yr lifetime (Ref. 8). One leading candidate is AZ93, a commercially available product of AZ Technology, Huntsville, Alabama. AZ93 is an inorganic white paint composed of a zinc oxide pigment in a potassium silicate binder and has significant space flight heritage. Using AZ93 on composite substrates requires AZ Technology's MLP-300 primer system as an interface layer.

The cost of AZ93 is substantial and of major concern for the technology demonstration program. Coating the 2- by 3-m Second Generation RDU panel with the currently preferred AZ93 ceramic coating could cost a large fraction of the technology demonstration program budget. A strategy to use a less expensive coating with similar infrared emittance to demonstrate the essential features of the Second Generation RDU was pursued, while retaining AZ93 as the preferred flight coating. One possibility given

the relatively short duration of the test program and lack of ultraviolet (UV) radiation in the test plan is to use a low-cost polyurethane coating instead. Hence, polyurethane coatings such as LORD Aeroglaze A276 are of interest to the test program. However, they may thermally decompose and/or outgas at the operating temperatures of interest (Ref. 9), so a survey was undertaken to determine the effects of thermal exposures similar to those expected during Second Generation RDU testing on Aeroglaze A276.

Normal operation of the Second Generation RDU will occur in vacuum at temperatures between 370 and 430 K. The total operating time of the Second Generation RDU will be determined by the yet to be established operating procedures but will likely be between 500 and 750 hr and not exceed 1500 hr. The testing to map the heat rejection performance of the panel will be completed within 500 hr. With these needs in mind, the first test of Aeroglaze A276 was conducted to determine if the coating undergoes a loss of material in vacuum. A standard test for total mass loss (TML) and collected volatile condensable materials (CVCM) was utilized.

Initially, the Second Generation RDU will be operated at low temperature in air as part of the installation of the unit into the vacuum chamber and checkout of the systems. However, it may be exposed to temperatures up to the nominal operating temperature of 400 K during installation if a full power test is required prior to evacuating the chamber. The acceptance testing may also require elevated temperature operation in air at a temperature up to 400 K. The second test of Aeroglaze A276 determined if an exposure in air at the nominal operating temperature of 400 K would result in gross discoloration (yellowing) and loss of material through volatilization or oxidation.

The emissivity as a function of exposure time was also examined to determine if the coating emissivity was constant or would be a time-dependent variable during testing of the Second Generation RDU.

Manufacturing of the Second Generation RDU will require an elevated temperature cure for the epoxy that will be used to join the pieces of the assembly together. This thermal exposure may also be used to ensure complete curing of the coating and outgassing of any remaining volatiles. The effects of such an exposure upon the emissivity of the coating were evaluated to determine when the coating should be applied during manufacture.

Experimental Procedure

Coating Application

LORD Aeroglaze A276 coating and thinner were obtained by Material Innovations Inc. (MII) from LORD as part of the Second Generation RDU technology demonstration program. The coating was sprayed onto one side of eight fully consolidated K13D graphite, EX-1522 epoxy (Gr/Ep) coupons approximately 5 by 5 cm. The spraying was done in two batches of four coupons. The manufacturer's suggested mixing ratio, thinning ratio, and spraying procedures were used (Ref. 10) to approach the desired coating thickness of 0.050 to 0.075. Following coating, the eight coupons were sent to NASA Glenn Research Center (GRC) for testing and evaluation.

Because of observed problems with spraying the coating, MII continued development of the spraying process. Two additional coupons with perceived improved coatings were supplied to NASA GRC for emissivity testing. One was in the as-received condition like the first eight coupons, while the second had been given a 450 K cure for 3 hr in air.

Total Mass Loss and Collected Volatiles Condensable Materials Testing

The first batch of four coupons determined the mass loss near 400 K, the intended operating temperature of the Second Generation RDU. Testing occurred at NuSil Silicone Technology, Carpinteria, California, in accordance with ASTM E 595 (Ref. 11).

The coating was scraped from the epoxy and crushed to make the sample. The sample was placed in a special furnace chamber, which had an exit port directed towards a cooled plate that collected condensable gases. Both the furnace and a condenser plate were located within a single environmental chamber. The sample was preconditioned by exposing it to air with 50 percent relative humidity for 24 hr prior to testing.

The sample was raised to a temperature of 398 K while under a pressure of <6.7 mPa for 24 hr. The volatile components generated by heating escaped through the exit port. Any volatiles that were condensable at 298 K were collected on the collector plate, which was maintained at 298 K. Following testing, the remaining sample was conditioned in 50 percent relative humidity air for 24 hr. The collector plate and sample were weighed before and after the test to determine the percentage weight change, TML, and CVCM from outgassing in a vacuum environment.

Thermal Exposures

The second batch of four coupons was used to evaluate the effects of thermal exposure on the coating. Emphasis was placed upon thermal exposures that were likely to be encountered during the testing of the Second Generation RDU rather than a comprehensive test matrix.

Three thermal exposures were conducted. The first simulated the exposure of a heated Second Generation RDU panel in air during acceptance testing and installation. A coupon was placed in a box furnace preheated to 400 K. This is the nominal operating temperature for the Second Generation RDU that will be used for acceptance testing and system checkouts. A static air atmosphere was used for the environment. The sample was removed after 74 hr and air cooled to room temperature.

The second and third thermal exposures were conducted in a vacuum furnace with a maximum chamber pressure of 4.3 mPa. The coupons were placed in the vacuum chamber one at a time, the chamber evacuated, and the coupons heated to 450 K within 30 min. The temperature was chosen as a conservative maximum temperature for any off-nominal testing beyond current plans. The only difference between the second and third thermal exposures was duration. The second test lasted 50 hr and the third test lasted 504 hr.

The coupons used in the first and third thermal exposures were weighed using an Ainsworth AA-160 analytical balance before and after exposure to determine their change in mass.

In addition, one coupon in the third batch of samples had a 3-hr thermal exposure at 450 K, in air, as part of its curing process. This exposure represents one possible cure for the adhesives that will be used to join the parts of the Second Generation RDU panel. Such a thermal cycle would also act as a final cure for the coating to ensure that complete curing is achieved prior to testing of the Second Generation RDU. This sample was also evaluated against the corresponding as-received room temperature cure specimen provided in the third batch.

Two uncoated Gr/Ep samples were exposed for 100 hr to estimate the weight change of just the Gr/Ep substrate during the thermal exposures. The first sample was exposed in air at 400 K, and the second sample was exposed in vacuum at 450 K.

Emittance Measurements

Emittance measurements were conducted at NASA GRC using a Gier-Dunkle DB-100 infrared reflectometer. The Gier-Dunkle infrared reflectometer illuminates the sample hemispherically utilizing a black body cavity heated to approximately 325 K and detects the reflected signal at a near normal angle. Because the Second Generation RDU will not be illuminated, solar absorptance was not measured. From the first two batches of samples, two coupons in the as-received condition (one from each batch) and one coupon for each of the three thermal exposures were evaluated. Both samples from the third batch were also evaluated. Six repeats were performed on the coupons at various locations and averaged.

Results

Appearance

Figure 1 presents photographs of the coating in the as-received and thermally exposed conditions. For reference, a background of standard white copy paper was used for all the pictures.

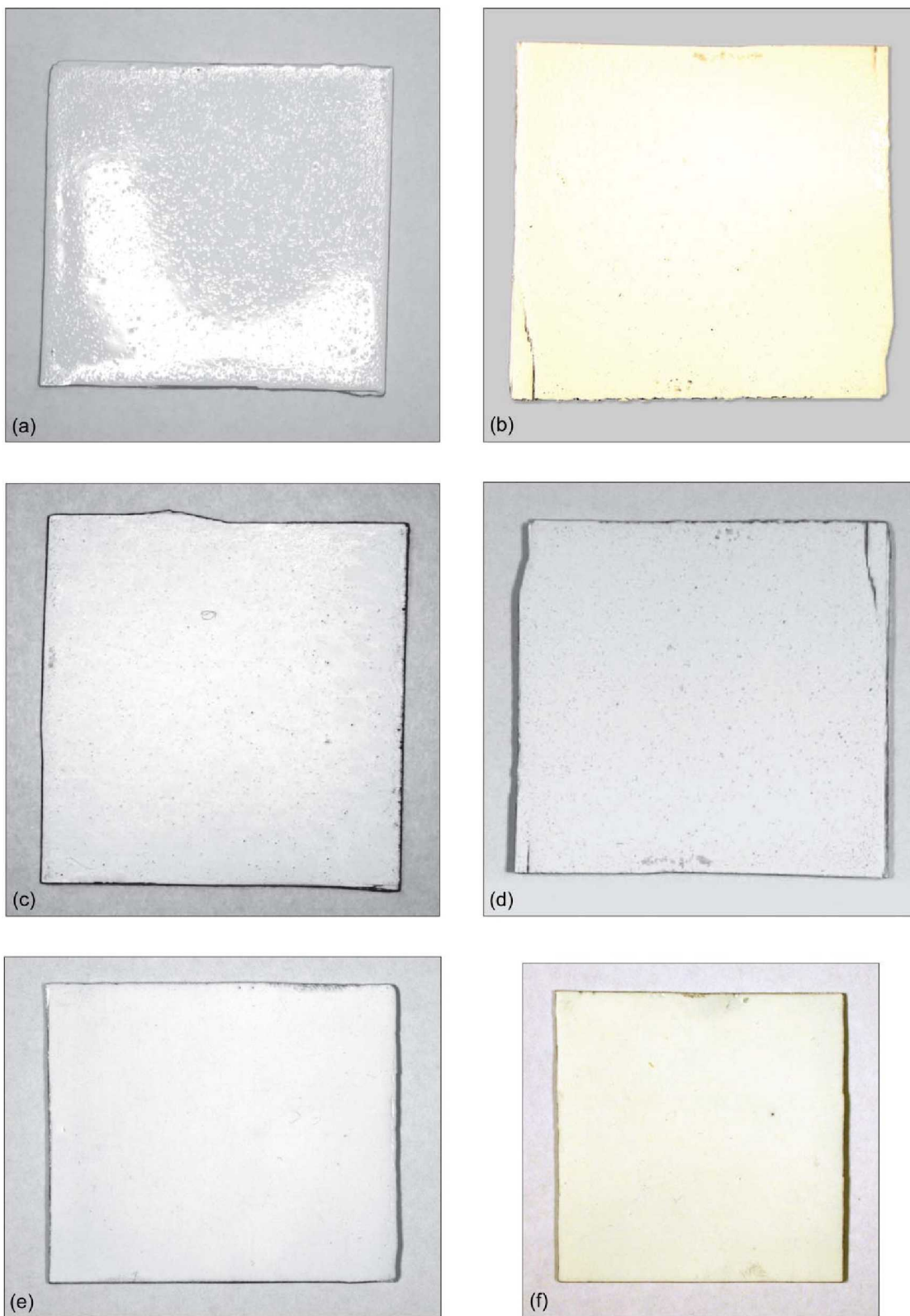


Figure 1.—Coating appearance. (a) As-received. (b) 400 K/74 hr/air. (c) 450 K/50 hr/vacuum. (d) 450 K/504 hr/vacuum. (e) As-sprayed (improved sprayed technique). (f) 450 K/3 hr/air (improved sprayed technique).

The as-received coupons from the first two batches had a rough surface with noticeable peaks and potential bubbles in the coating as shown in Figure 1(a). Consultation with MII indicated that the Aeroglaze A276 coating sprayed in unusually large droplets that approached globules. As a result, the thickness as measured by MII after coating was 0.25 mm rather than the desired 0.05 to 0.08 mm thickness. The coating did not fully cure at room temperature before shipping. The surface may therefore represent an intermediate condition between as-sprayed and fully cured.

The product information sheet provided by LORD indicated that coatings greater than 0.13 mm can exhibit bubbling (Ref. 10). This appears to have occurred for the samples in the first two batches. Gloss was not measured, but the coating appeared to be consistent with the value provided in the product information sheet.

Exposure to air at 400 K for 74 hr produced three noticeable changes. The surface of the coating became smoother, the gloss of the coating decreased, and the color yellowed slightly, as shown in Figure 1(b). The yellowing was not severe and is generally not noticeable unless it is compared to a white reference. The glossy surface of the sample also became a semigloss surface.

Three things may have occurred during the thermal exposure to smooth the coating surface: (1) the coating completed curing, (2) the coating underwent viscoplastic creep, and (3) trapped gas diffused to the surface and was lost. All three phenomena could lead to a flatter surface. It is uncertain which mechanisms were operative during the thermal exposure.

Exposure in vacuum at 450 K did not produce any noticeable change in color, as shown in Figures 1(c) and 1(d). As with the coupon exposed in air, the surface became smoother after the thermal exposure. It is believed that the same mechanism(s) that caused this change for the air sample caused this change for the vacuum samples. For these coupons, the surface finish changed from glossy to matte with little if any evidence of gloss remaining.

The spraying problems, excessive coating thickness, and bubbles led MII to thin the coating more than the manufacturer's recommendation to eliminate these problems. Success was achieved, as can be seen in Figures 1(e) and 1(f), by reducing the coating thickness to 0.08 mm and eliminating all bubbles. The coating remained white and glossy in the as-sprayed condition.

Following thermal exposure at 450 K for 3 hr in air, the coating underwent a very slight yellowing. The degree of yellowing was judged in a side-by-side visual comparison to be less than the yellowing experienced following exposure at 400 K for 74 hr in air. This indicates that the yellowing is likely progressive and dependent upon both time and temperature. The gloss appeared to be comparable to the as-sprayed specimen and much greater than the specimen exposed at 400 K for 74 hr.

Mass Loss

The results obtained by the method outlined by ASTM E 595 (Ref. 11) were that the Aeroglaze A276 coating alone has a TML of 0.57 percent and a CVCM of 0.01 percent. The CVCM value is at the limit of detectability, and the value reported is likely an upper limit for the sample.

Table I lists the mass losses for the specimens exposed at NASA GRC. The measured weights and weight changes presented are for the entire coupon (coating plus substrate).

TABLE I.—WEIGHT LOSS OF THERMALLY EXPOSED COUPONS

Sample	Initial weight, g	Final weight, g	Weight change, g	Weight change, %
Coated, 400 K, 74 hr, air	2.9526	2.9267	−0.0259	−0.88
Coated, 450 K, 504 hr, vacuum	3.2296	3.0979	−0.1317	−4.08
Uncoated, 400 K, 100 hr, air	2.6433	2.6428	−0.0005	−0.02
Uncoated, 450 K, 100 hr, vacuum	2.7576	2.7482	−0.0094	−0.34

Emittance

The results of the emittance measurements are shown in Table II. For comparison the emittance of a similar uncoated Gr/Ep sample measured at NASA GRC previously is also presented. Based upon current design models an emittance of 0.8 is needed to meet the heat rejections goals of the Second Generation RDU. The uncoated Gr/Ep face sheet cannot meet this requirement, but all of the coated coupons appreciably exceed the requirement.

TABLE II.—LORD AEROGLAZE A276 EMITTANCE MEASUREMENTS

Condition		Average	Standard deviation	Change from as-received, percent ^a
Uncoated Gr/Ep		0.771	0.020	–15.9
As-received	Batch 1	0.932	0.001	
	Batch 2	0.903	0.003	
As-received average		0.917	0.003	
450 K, 50 hr, vacuum		0.885	0.003	–3.5
450 K, 504 hr, vacuum		0.869	0.001	–5.3
400 K, 74 hr, air		0.901	0.005	–1.8
As-received (improved spraying)		0.893	0.006	
450 K, 3 hr, air (improved spraying)		0.896	0.002	0.4

^aChange from as-received refers to the as-received average of the first two batches or the as-received value for the improved spraying sample for the third batch.

To compare the four emittance values from the initial eight samples statistically a one-way analysis of variance (ANOVA) was conducted. The results showed that there was a statistically significant difference between the average emittance values for the four conditions. A Student-Newman-Keuls (SNK) analysis (Ref. 12) was conducted to determine which averages were statistically significantly different. The SNK test showed that the as-received and the air exposure samples had statistically equivalent emittance values. The 50-hr vacuum sample had a lower emittance, and the 504-hr vacuum sample had the lowest emittance $[(\epsilon_{\text{As-received}} = \epsilon_{74 \text{ hr/air}}) > \epsilon_{50 \text{ h/vac}} > \epsilon_{504 \text{ hr/vac}}]$.

The improved spraying technique may have altered the emittance, so these two samples were treated separately. A comparison of the averages of just these two coupons using a t-test showed that the averages of the as-sprayed and 450 K/3 hr/air samples were statistically equivalent. This is consistent with the results of the first two batches. An ANOVA of the as-received samples from the first two batches and the two samples from the third batch showed a statistically significant difference in the means. Comparisons of the three data sets showed that the as-received samples from the first batch had a greater average emittance than both samples from the third batch, indicating the increase in thinner yields a slight decrease in emittance. It is not clear with the limited number of samples if this difference falls within the normal scatter of the coating's emittance or if the modified process is affecting the emissivity.

The average emittance values for the as-received coupons from the first two batches and the coupons exposed in vacuum are plotted in Figure 2. With only three data points it is impossible to truly determine if the decrease in emittance is following a linear, power law, or parabolic decrease. It appears that the rate of change in the emittance is time dependent and decreasing with time, but additional tests would be needed to determine the true form of the trend.

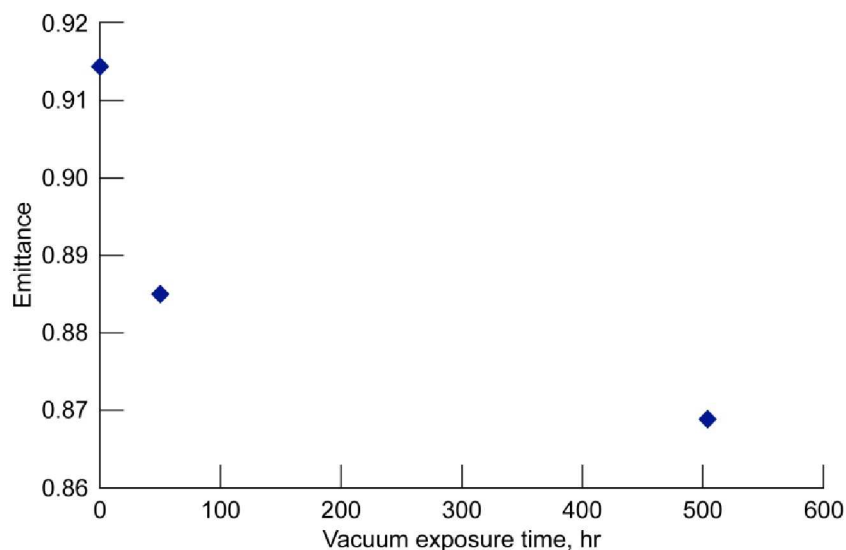


Figure 2.—Change in emittance with time during thermal exposure at 450 K in vacuum.

Discussion

Spray Coating

The initial coating was too thick and suffered from bubbles and other problems. By increasing the amount of thinner, the coating was able to be better atomized and sprayed. The result was a thinner coating that approached a mirror finish and that met the requirement for a 0.05- to 0.08-mm-thick coating. There was a slight decrease in the emittance of the coating using this technique. The improvements in the coating achieved with the new spray technique was judged to be worth the emittance penalty since the emittance value for the as-received sample was still well above the minimum required value of 0.8.

Mass Loss

The TML as measured by the E 595 test method confirmed that the Aeroglaze A276 coating was a low outgassing (<1 percent TML) coating in vacuum. The results of the furnace exposures indicate that more volatiles were released during longer exposures since the mass loss of the coupons increased with time. The 450 K vacuum exposure also accelerated the outgassing of the painted coupons, which led to a greater weight loss.

The published properties of Aeroglaze A276 list a TML of 0.99 percent and a CVCM of 0.08 percent following a 15-day cure at room temperature (Ref. 10). The samples sent to NuSil exceeded this cure time before they were tested, so they were representative of a fully cured coating. The results obtained by this study indicated lower but similar TML and CVCM values. It is likely that the manufacturer's published values represent a conservative upper limit, so it is believed that the values are consistent.

The furnace exposure results show that up to a 4 percent change in weight occurs in the coated coupons while the uncoated specimens had much lower weight losses. Extrapolating the weight loss for the uncoated vacuum sample linearly to 500 hr gives a conservative weight loss for the substrate of 1.7 percent. This estimate is conservative because it does not take into account the protection given the substrate by the coating for the coated sample, it does not take into account the larger surface area and shorter diffusion paths for volatiles to reach the surface and escape from the substrate, and it assumes the worst case linear weight loss behavior rather than parabolic or other behavior commonly seen in diffusion rate controlled processes.

The data from this study was compared to data from Shin who has measured thermally induced weight losses on similar epoxies (Ref. 13, personal communication). Thin specimens pressed between metal plates were tested at 398 K in a pressurized helium gas environment. The weight loss of a thin sample with only the edges exposed was about 0.35 percent in 4 mo. Since the coupons are coated on one side and laying on a flat surface, this test is likely indicative of the weight loss that would be experienced by the Gr/Ep substrate. During the duration of these tests weight losses of less than 0.1 percent would likely be experienced by the substrate. Vacuum would tend to promote diffusion by reducing the partial pressures of the evolved gases at the surface, so a larger weight loss would be expected. This expectation and the results from Shin are consistent with the observations reported in Table I.

The much lower weight loss for the uncoated specimens indicates that the majority of the measured weight loss comes from the coating. An estimate was made using the measured thicknesses of the coating and the Gr/Ep substrate and making two assumptions. First, the density of the coating was assumed to be 0.63 g/cm^3 based upon subtracting the volatile density from the total density of the liquid coating (Ref. 10). This value is slightly lower than the value of 0.64 g/cm^3 determined from the solids content. The second assumption was that the substrate lost between 0.1 and 1.7 percent during the 504-hr exposure based upon the weight losses observed by Shin and the uncoated specimen, respectively. Using these measurements and assumptions, the coating weight loss was estimated to be between 13 and 19 percent. While greater than desirable, the value indicates that there will still be a 0.062-mm- thick coating on the Second Generation RDU after 504 hr of testing at 450 K if the initial coating thickness is 0.08 mm. This value is within the specified limits for the coating thickness and therefore, is deemed acceptable.

The concern with using a polyurethane-based coating such as Aeroglaze A276 compared to the preferred ceramic-based coating with a higher temperature capability is the potential for the coating to thermally decompose when heated. Such heating could decrease the emittance. The coupon exposed in air showed some slight yellowing probably caused by minor oxidation but no change in emittance. The coupons exposed in vacuum and the TML testing showed some volatiles were generated, including adsorbed water and hydrocarbons. The volatiles likely included alcohol and perhaps carbon dioxide from the pyrolysis of the polyurethane (Ref. 9). The amount appears to increase with time which would be expected if the loss of material is controlled by diffusion, but the loss was still acceptably low after 504 hr in vacuum at 450 K for the Second Generation RDU application. As such, the observed mass losses did not disqualify Aeroglaze A276 from use on the Second Generation RDU.

Emittance

Coating the Gr/Ep substrate with Aeroglaze A276 increased the emittance from 0.771 for the uncoated material to 0.918 for the coated material. This represented an increase of 19 percent. To reject the amount of heat required by the Second Generation RDU, a requirement was established for an emittance of at least 0.8 based upon current design models, and a higher emissivity was desirable to enhance heat rejection. The coating provided a clear and significant benefit in this application and will in fact be needed to meet performance requirements.

The initial emittance was also similar to the preferred coatings for lunar applications, such as AZ93. As such, the initial heat rejection performance of the Second Generation RDU should be similar to the potential lunar outpost radiators. The decrease in emittance with time is a potential problem. The testing indicates that during the portion of the test plan to map the performance of the radiator panel a decrease in emittance of 4 percent will occur. However, the value after 504 hr at 450 K in vacuum is still similar to the emissivity of AZ93, so the performance of the Second Generation RDU should be similar to future lunar radiators. The differences will be taken into account through future modeling.

A change in the emittance was observed with the improved spraying practice. Manufacturing considerations for producing a suitably thin coating on the Second Generation RDU required making the change in the spray technique. The slight decrease in the as-received emittance relative to the as-received average emittance of the first two batches was not considered to be an issue for the Second Generation RDU since the emittance was still well above the required value of 0.8. It is expected that the improved spraying

technique will yield a surface that will be similar to the first two batches when thermally exposed, and that the decrease in emittance will be similar. The emittance of the coating following thermal exposure during testing will still allow the Second Generation RDU to meet the heat rejection requirement. The results further indicate a need to measure the emittance of the coated Second Generation RDU panel before, during, and after testing.

For the conditions expected during the testing of the Second Generation RDU the Aeroglaze A276 coating is expected to experience only a small change in emittance. The emittance values should remain sufficiently high to meet heat rejection requirements. While it is recognized that Aeroglaze A276 would not be a suitable coating for flight hardware, it provides similar emittance performance at a much lower cost making it well suited for the Second Generation RDU technology demonstration program.

Conclusions

Aeroglaze A276 is a reflective, low-outgassing, polyurethane coating produced by the LORD Corporation. It produces a gloss white finish with a high infrared emittance. The emittance drops from 0.918 for the as-received condition to a minimum of 0.869 following a 504-hr exposure at 450 K in vacuum when utilizing the manufacturer's mixing ratios. Air exposure does not affect the emittance but does induce limited yellowing of the coating. Thinning the coating to improve spraying causes a 2.7 percent decrease in the as-received emittance but does not affect the observed changes from short term thermal exposure in air.

Testing of samples in vacuum and in air shows that minimal changes occur in the emittance of the coating for durations consistent with the planned Second Generation Radiator Demonstration Unit (RDU) testing. These changes indicate that monitoring the emittance of the coating during Second Generation RDU testing is prudent to provide the best correlation between test data and modeling. However, the overall changes in emittance are sufficiently small that they are unlikely to impact the test results. Aeroglaze A276 is a suitable coating for the Second Generation RDU.

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14. ABSTRACT A lunar outpost will require electrical energy. One potential source is fission surface power where heat from a reactor is converted into electricity utilizing an energy conversion system, and waste heat will need to be rejected from the system. The Second Generation Radiator Demonstration Unit is a technology demonstration unit leading towards operational radiators. To approximate the infrared emittance of the lunar outpost radiators, a low-cost coating compatible with the test conditions was sought. LORD Aeroglaze A276 has a similar emittance, but its performance in air and vacuum at the desired operating temperatures was unknown. This study determined that the emittance remained above 0.86 for all conditions tested and that LORD Aeroglaze A276 is a suitable surrogate coating for the Second Generation Radiator Demonstration Unit.					
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